

^{13}C NMR of solid materials obtained with magic angle spinning but without cross polarization can selectively detect mobile components dispersed within the solid, such as solanesol in tobacco. An attempt was made to observe menthol on cellulose acetate by this method. Significant signals were observed from the cellulose acetate in addition to a very broad background signal centered about the aromatic region. The ^{13}C MAS spectra of cellulose (cotton linters) also exhibited signals from the anhydroglucose units of the polymer but no background was observed. The MAS spectrum of cellulose differs from the CP/MAS spectrum in that the narrow line components attributed to crystalline cellulose are absent as well as both the narrow and broad lines for C-4. Several different cellulose samples were examined including the insoluble residue from tobacco fractionation, raw cotton, cotton linters, Sigmacell, Avicel, and acetobacter xylinum pellicle. The most amorphous of these samples, the tobacco cellulose, exhibited the strongest signals while the most crystalline, acetobacter, exhibited no signals at all. The method appears to be selective for either the amorphous regions, end group anhydroglucose units, or both. A ^{13}C T₁ measurement showed that the C-6 (hydroxymethyl) signal decays significantly more rapidly than the remaining signals, reflecting a surprising degree of motional freedom. The proton MAS NMR spectra of the cellulose samples was also observed but only a very broad signal from the rigid lattice protons was detected. A pulse sequence was encoded that employs a Hahn echo to suppress the rigid lattice signals. Each of the cellulose samples that have been observed with the spin echo pulse sequence have shown a relatively narrow proton signal which apparently corresponds to the same cellulose component detected in the ^{13}C MAS spectra.³

New data were obtained on the kinetics of the cooked flavor reaction which was monitored by ^{13}C solution NMR. By adjusting the solution concentrations and carefully adjusting the spectrometer, spectra were obtained with better signal to noise and stability than previously obtained. The relative concentrations of glucose, fructose, glucosyl amine, diglucosyl amine, and deoxyfructosazine were followed over a two day period. The data are being analyzed by John Lephardt.³

REFERENCES

1. W. N. Einolf, Notebook 8264.
2. D. F. Magin, Notebook 8231.
3. J. Wooten

W. N. Einolf

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MS (D. Magin, N. Einolf)

LTF-IIA cigarettes were made with each of the following components at the 10% level (as the β -D-glucopyranoside): phenol, hydroquinone, menthol and 6,7-dihydroxycoumarin. Each cigarette type was smoked, the TPM extracted with dimethylformamide (DMF), and an aliquot of the DMF solution was derivatized with BSTFA/1% TMCS. The TMS derivatives of the aglycone and intact β -D-glucoside were quantitated by GC using 4-t-butylphenol as an internal standard. The delivery of aglycone and glucoside in TPM are as shown, and are given as percent of available glucoside in the filler.

	<u>% as aglycone</u>	<u>% as glucoside</u>	<u>% Total Transfer</u>
phenol	1.76	8.90	10.66
hydroquinone	6.79	2.70	9.49
menthol	0.72	10.5	11.2
6,7-dihydroxycoumarin	3.11	0.14	3.25

These cigarettes were unfiltered. In separate experiments, filters were attached to the LTF-IIA cigarettes containing menthylglucoside, and the resulting TPM gave a 0.27% menthol and a 7.5% glucoside delivery.¹

GC/MS samples were analyzed at the request of D. Williams, J. Paine, D. Howe, T. Crews and R. Carpenter.²

The Wiley Data Base transfer is complete to our SS 200 data system. We are currently rebuilding the Wiley library, however, since it required more disk space than was originally allotted.²

NMR (J. Wooten, R. Bassfield, J. Campbell)

¹³C CP/MAS spectra were obtained on six burley tobacco samples obtained from Walter Hempfling. Each sample has been extracted by various methods to remove protein. Difference spectra were obtained with a spectrum of normal burley tobacco revealing the types of material removed from each sample. More significantly, the CP/MAS spectra showed the extent of protein remaining. Signals in the high field aliphatic region indicate that proteins rich in amino acids with neutral, non-polar side chains are least affected by all the extraction methods. One detergent extracted sample exhibited strong signals from the detergent in its MAS spectrum obtained without cross polarization. Although strongly retained by the tobacco, the detergent remains very mobile and displays narrow signals in the tobacco which are very similar to the signals observed in its D₂O solution ¹³C spectrum.³

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